5. TEST AND MATERIALS STANDARDS

A. Modification of Engineering Materials for Heavy-Vehicle Applications

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Objectives

- Organize an international cooperative research program on an integrated surface modification technology under the auspices of the International Energy Agency (IEA).
- Design and identify surface features and patterns that can achieve friction reduction and enhanced durability for heavy-duty diesel engine components.
- Develop the understanding and appropriate models to explain texturing effects on frictional characteristics. Develop appropriate thin films and coatings to achieve a synergistic and complementary relationship with texturing to enhance performance.
- Discover and develop the surface chemistry for protecting the films and coatings that work in synergy with the textures.

Approach

- Review the literature database to compile the current practices on surface texturing, thin films, and coatings. Understand the cause and effects of the beneficial attributes of texturing used in different applications.
- Based on the current understanding, design various potential surface textures that might enhance performance while minimizing deleterious effects.
- Develop and explore various ways to fabricate these surface textures on appropriate surfaces. Compare cost and effectiveness ratios for these fabrication techniques.
- Develop a test methodology to measure the effects of these textures on friction and wear reduction and develop mapping techniques to define the limits of applicability in terms of operating parameters such as load and speed for a given materials pair.

- Conduct research to develop an integrated system approach to combine the best practices in thin films, coatings, and surface chemistry for performances unrealizable by an individual approach alone.
- Concurrently, organize an international cooperative research program under the auspices of the IEA to pool resources and share this energy conservation technology worldwide.
- Under IEA annex IV, seek approval from the IEA executive committee and organize a U.S. national working group centered at NIST to provide industrial input and exchange information with other national groups.

Accomplishments

- Organized a special symposium on an integrated surface modification technology during the ASME Joint Tribology Conference on October 27–29, 2003. Speakers from Finland, Sweden, the United Kingdom, and Israel described activities in their countries; and DOE, NIST, Argonne National Laboratory, Oak Ridge National Laboratory, and the National Science Foundation described various research activities in the United States. Caterpillar and others provided an industrial perspective and information on industrial needs. More than 100 people attended the 1-day session (40–50% of the total conference attendees).
- Developed a proposal on forming an annex IV on integrated surface modification technology. Attended the IEA executive committee meeting held in Oakland, California, on October 22, 2003, and presented the detailed plan to the executive committee.
- Using a pin-on-disk test, compared the friction reduction characteristics of various geometries under the same surface coverage. These features were prepared using photolithography and mechanical scribing.
- Investigated a test design that uses a disk with four different textures separated by untextured areas as an efficient means to evaluate and compare several textures in a single test.
- Found that a surface pattern of elliptical features reduces the coefficient of friction under mixed-film lubrication conditions compared with a surface without texture, but only when sliding was perpendicular to the long-ellipse axis.

Future Plans

- Organize a U.S. national working group under the IEA banner to conduct joint research and provide information for international exchange.
- Examine next the issue of surface texture density and depth of features on elliptical features.
- Evaluate the test data to determine which feature and pattern characteristics, either singly or in combination, are most critical with respect to their effect on the coefficient of friction. Based on the outcome, develop parametric equations relating feature and pattern characteristics to the coefficient of friction.

Introduction

Frictional losses are inherent in all mechanical mechanisms that have contacting elements in relative motion. Reduction of such frictional losses arising in the mechanical systems of trucks and automobiles alone could result in substantial energy savings. Recent studies have shown that one means of reducing friction in sliding contacts is by introducing an appropriately designed surface texture on the bearing surfaces. This finding offers a significant opportunity for reducing friction losses in sliding contacts. The initial studies have shown that the effect depends strongly on pattern design, contacting materials, and lubricant properties. A full understanding of these dependencies has yet to be developed. The objective of this project is to identify critical pattern features that control friction under a broad range of contact conditions and develop models to explain these relationships. In the few months that this project has been under way, test procedures have been developed and a number of surface texture patterns have been evaluated using a laboratory pin-on-disk test.

Approach

The effects of different surface texture patterns were evaluated with a conventional pin-on-disk tribometer, illustrated schematically in Figure 1. Load and sliding speed were varied to control the contact conditions. Tests were conducted using mineral oil without additives, having a viscosity of 2.73×10^{-5} m^2/s @ 40ºC. Pin-and-disk specimens were fabricated from cold-rolled 1017/1018 steel and 304 stainless steel. Friction force was monitored and recorded during the test. Two methods were used to produce surface textures on specimens. One was a photolithography method employing a mask fabricated with features of the size, shape, and spacing for the desired texture pattern. A photoresist was applied to the polished ($R_a \approx 0.01$ μm) test surface and exposed to light through the mask. After development, the specimen was electrolytically etched sufficiently to produce texture features of the desired depth. The other method was by mechanical scribing. A scribe (Rockwell "C" indenter) was held without rotation in the spindle of a small milling machine and pressed against the specimen mounted on the milling machine table. By controlling the table motion and application of the scribe, either indentations or grooves could be produced. After scribing, the specimen was polished flat to remove the raised lips around the scribed features.

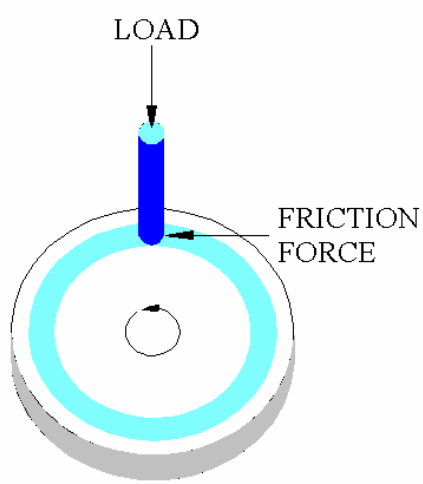
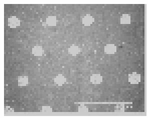
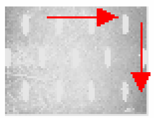


Figure 1. Schematic of pin-on-disk test configuration.

Results

Table 1 gives the characteristics of two different patterns, circular dimples and ellipses, that were generated on pin specimens by the photolithography method; note that the feature area-density and feature depth were the same for both patterns. To assess the effect of orientation, two sliding directions, indicated by arrows in Table 1, were investigated for the ellipses. Test results for the patterns are shown in Figure 2 and compared with a polished pin without texture. The pin-and-disk materials in this example were cold-rolled 1017/1018 steel. To control lubricant film thickness, sliding speed and applied load were varied in the range of 0.023 m/s-0.23 m/s and 1 N-35 N, respectively. In Figure 2, the coefficient of friction is plotted as a function of the Sommerfeld number (viscosity × speed/load). There is little difference in friction behavior among the tests except for the elliptical pattern with sliding perpendicular to the direction of the long-ellipse axis. In this case, the coefficient of friction remains low for small-Sommerfeld-number values, while the other tests exhibit a substantial increase in coefficients of friction. Apparently, the presence of the elliptical texture pattern for

Table 1. Characteristics of two surface textures prepared by photolithography method

	Pattern & Sliding dir.	Dimension (μm)	Depth (μm)	Pitch (μm)	Area of a dimple (μm^2)	Area density (%)
Circle		150	8	500	17671	7
Ellipse		300/75	8	500	17671	7

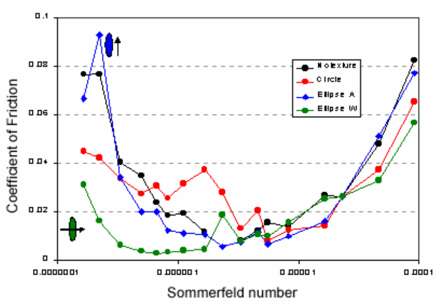


Figure 2. Pin-on-disk test results for different surface textures produced on a pin surface by the photolithography method compared with results for a pin surface without texture.

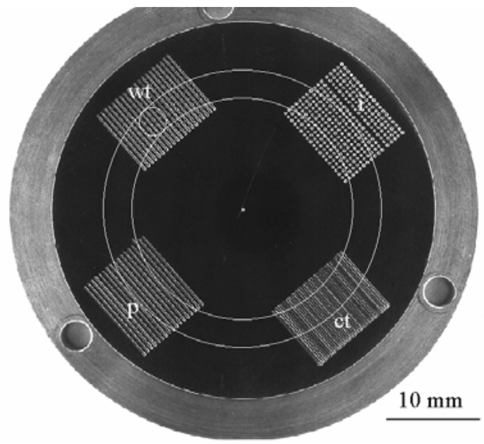


Figure 3. Disk specimen with four different scribed texture patterns.

this sliding direction results in a substantial extension of the mixed-lubrication regime. Complete understanding of the effect, however, will require additional investigation.

As a second example, a series of tests was conduced on a 304 stainless steel disk (Figure 3) scribed with four different patterns: (1) grooves (wt) at a spacing of 111 μm, lying approximately in the radial direction; (2) grooves (p) with the same size and spacing as (wt) oriented approximately parallel to the direction of sliding; (3) grooves (ct) similar to (wt) but spaced at 54 μm; and (4) a pattern of dimples (i) spaced at 111 μm. Each pattern covered approximately the same area and was separated by approximately the same circumferential length of untextured surface. The path of the cylindrical pin and its relative contact size are indicated in Figure 3. The variation in coefficient of friction for three revolutions of the disk is shown in Figure 4 for a test conducted at an applied load of 15 N and sliding speed of 10 mm/s. The section of the plot corresponding to the location where the pin passed over each pattern is identified in the figure. In each case, the presence of the texture resulted in an increase in coefficient of friction compared with the intervening untextured surface. Among the textures, the pattern of

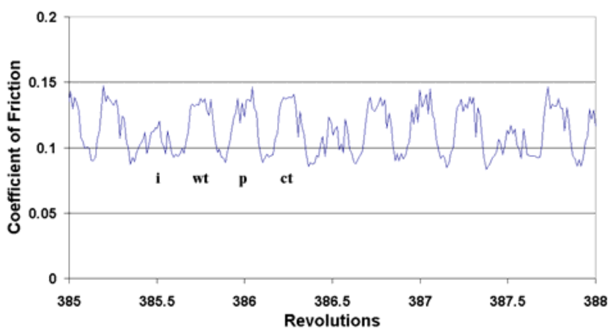


Figure 4. Coefficient of friction behavior for three revolutions of the disk in Figure 3.

indentations (i) gave a smaller increase in coefficient of friction. Similar results were obtained for other loads in the range of 2 N to 20 N.

Conclusions

Consistent with the findings of other investigators, this research found that surface texturing can reduce the coefficient of friction, offering a promising means for reducing energy consumption. However, depending on the contact conditions, a given texture pattern also can produce an increase in coefficient of friction or have no net effect on coefficient of friction. It is clear that additional investigation will be required to identify the critical texture characteristics that affect friction under a given set of conditions.

B. Implementing Agreement for a Programme of Research and Development on Advanced Materials for Transportation Applications

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Contractor: Oak Ridge National Laboratory, Oak Ridge, Tennessee
Prime Contract No:DE-AC05-00OR22725

Objectives

- Facilitate the integration of new technologies into the diesel engine community by implementing research that validates the applicability of this technology to improve material properties while maintaining acceptable life-cycle costs.
- Promote commercialization of new materials technologies by developing standard testing and characterization methods in conjunction with national and international standards communities.

Approach

- Define and implement research under the International Energy Agency (IEA) Implementing Agreement entitled "Implementing Agreement For A Programme Of Research And Development On Advanced Materials For Transportation Applications" (IA–AMT).
- Conduct major research themes as annexes under the current Implementing Agreement.
- Annex II Cooperative Program on Ceramics for Advanced Engines and Other Conservation Applications
- Annex III Cooperative Program on Contact Reliability of Advanced Engine Materials

Accomplishments

- Initiated new efforts (subtasks 11 and 13) under Annex II.
- Defined the scope of work for Annex III
- Annex II Cooperative Program on Ceramics for Advanced Engines and Other Conservation Applications.
- Issued an invitation to the United Kingdom from the Executive Committee of the Implementing Agreement to join the IA-AMT because of its interest in participating in Annex III.

- Held preliminary discussions with government officials from Canada concerning the initiation of a new annex on lightweighting of materials.

Future Direction

- Develop a plan for the lightweighting materials annex and present it to the Executive Committee for approval.
- Initiate a test plan for Annex III. Current participants are United States, Germany, Japan, and United Kingdom.

Introduction

The IEA was formed via an international treaty of oil-consuming countries in response to the energy crisis of the 1970s. A major objective of the IEA is to promote secure energy supplies on reasonable and equitable terms. The governing board of the IEA, which is composed of energy officials from each member country, regularly reviews the world energy situation. To facilitate this activity, each member country provides energy experts who serve temporary staff assignments at IEA headquarters. These staff, or the secretariat, support the governing board by collecting and analyzing energy data, making projections in energy usage, and undertaking studies on specialized energy topics. The governing board is also assisted by several standing groups, one of which, the Committee on Energy Research and Technology (CERT), encourages international cooperation on energy technology. Implementing agreements (IAs) are the legal instruments used to define the general scope of the collaborative projects. There are currently 40 active IAs covering research topics such as advanced fuel cells, coal combustion science, district heating and cooling, enhanced oil recovery, fluidized bed conversion, fusion materials, solar heating and cooling, pulp and paper, hydropower, heat pumping technologies, hybrid and electric vehicles, high-temperature superconductivity, wind turbines, and high-temperature materials. A complete listing can be found at the IEA website, www.iea.org.

This progress report summarizes recent activities in the IA-AMT. The objectives of the IA-AMT are (1) to identify and evaluate promising new processing and surface engineering technologies capable of improving materials performance in transportation systems and (2) to promote and implement pre-competitive development and verification of advanced characterization methods appropriate for advanced materials for transportation applications.

Approach

The collaborative research thrust areas (annexes) are defined by the Executive Committee of the IA-AMT. Their approach is to continually solicit ideas for new projects from (1) the technical leaders of the various activities, (2) recognized materials experts, and (3) special consultants. The IA-AMT currently consists of two active annexes: Annex II: Cooperative Program on Ceramics for Advanced Engines and Other Conservation Applications, and Annex III: Cooperative Program on Contact Reliability of Advanced Engine Materials.

Annex II currently consists of four active projects or subtasks (1, 11, and 13). The objective of Subtask 1 is to achieve a balanced exchange of technical information among the participants on high-performance ceramics for advanced engines and other conservation applications.

In Subtask 11, techniques for the measurement of thermal and mechanical fatigue are being examined. National efforts in Japan and the United States focus on the development of procedures for evaluating the mechanical fatigue behavior of silicon nitride ceramics using either uniaxial flexure or rotary bend test specimens. The national effort in Germany consists of the development of thermal fatigue procedures using the laser thermal shock equipment evaluated in Subtask 9.[1] The national effort in Sweden focuses on the evaluation of the fracture surfaces of specimens tested in Japan, the United States, and Germany. Subtask 11 also includes an international effort in which the thermal fatigue behavior of a single silicon nitride is compared with the mechanical fatigue data generated at a temperature that is the same as that at the fracture point in the thermal fatigue test. Germany is responsible for the thermal fatigue testing, and both Japan and the United States are conducting mechanical fatigue testing.

Subtask 13 includes a round-robin in which a selected silicon nitride material will be evaluated in burner rig and oxidation facilities located in Japan, Germany, and the United States. This effort is motivated by the results of recent ceramic gas turbine field studies conducted at both Rolls-Royce and Solar Turbines, which have demonstrated the deleterious effects of the gas turbine environment upon silicon nitride component lifetime. Specifically, recession of non-oxide ceramics such as silicon carbide and silicon nitride ultimately leads to excessive material loss, which increases the stress levels arising from both thermal and mechanical loading. Consequently, considerable emphasis has been placed on the development of environmental barrier coatings to act as a diffusion barrier to water vapor.

The effectiveness of environmental barrier coating systems is defined by (1) their ability to isolate the non-oxide ceramic from the water vapor and (2) their phase stability in the gas turbine environment. Both aspects can be evaluated from burner rig studies. Unfortunately, the results obtained from a given burner rig system depend upon a number of factors, including specimen geometry, gas flow/specimen configuration, temperature uniformity, and burner stability. The goal of this subtask is to address these issues by implementing a round-robin in which a selected silicon nitride material is evaluated in burner rig facilities located in Japan, Germany, and the United States.

Annex III, which was approved in July 2002, consists of two subtasks on contact reliability of advanced engine materials, including structural ceramics, composites, and nanostructured friction/wear coatings. Subtask 1 is an information exchange; Subtask 2 focuses on the development of standard test methods and procedures for determining the rolling contact fatigue resistance of advanced materials. The Executive Committee plans to invite the United Kingdom to join the IA-AMT because of its interest in participating in Annex III.

Results

Annex II

The final draft of the U.S. report for Subtask 11 was completed. The primary objective of this effort was to develop and verify techniques for the measurement of thermal and mechanical fatigue of structural ceramics. A major finding from this work concerned the excellent correlation between static and dynamic fatigue. The static fatigue data measured at 850ºC using the GS44 silicon nitride flexure specimens are shown in Figure 1 (circles). As discussed in ref. 2, the susceptibility of GS44 to time-dependent loss in strength was much greater at this temperature than the susceptibility observed at temperatures <700ºC. The associated strength degradation mechanism was attributed to softening of the intergranular phases, which ultimately led to viscous flow and separation of grain facets. Given sufficient time, this viscous flow was ultimately responsible for the formation of cavities within the intergranular phase. For example, Figure 10 compares the fracture

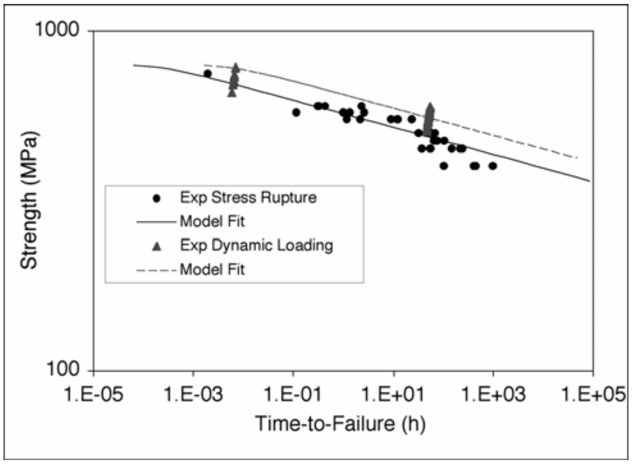

Figure 1. Comparison of static and dynamic fatigue data.

surfaces of two dynamic fatigue specimens; one tested at 30 MPa/s and other at 0.003 MPa/s. At 0.003 MPa/s, there was sufficient time for the cavitation to occur, as evidenced by the skeletal pattern of the intergranular phase outlining the interfaces between adjacent grains. It should be noted that similar behavior was observed for a hot-isostatically-pressed silicon nitride tested under tensile loading at temperatures above 1200°C.[3] In that study, however, the temperatures were sufficiently high to activate addition deformation mechanisms, including cavitation of the silicon nitride by a solution-precipitation process.

The experimental static fatigue data were subsequently fit to the generalized slow-crack-growth expression

$$[1-(S_f/\ S_i)^{N-2}] = (N-2)/2\ V(K_{IC})\ (Y\,S_i/K_{IC})^2\,(\sigma/S_i)^N\ t \qquad (1)$$

where Y is a constant, K_{IC} is the fracture toughness, $V(K_{IC})$ and N are the slow-crack-growth constants, V is the crack velocity, t is time, σ is the applied stress, S_i is the inert strength, and S_f is the strength after time, t. Failure occurs when $S_f = \sigma$.

The curve fitting process involved choosing values of S_i, N, and $V(K_{IC})$ that minimized the sum of the square of errors between predicted and measured lifetimes.

The fracture toughness at 850°C was assumed to be comparable to the room-temperature value reported by the vendor (8 MPa $m^{1/2}$). The resulting values of the crack growth parameters estimated using this iterative procedure are provided in Table 1. The solid line in Figure 1 illustrates the model prediction. Note that the value of $V(K_{IC})$ is relatively low. One might expect that it should be comparable to the speed of sound, given that it is associated with catastrophic fracture. However, this parameter actually represents the velocity occurring at the intersection of the Region I slow-crack-growth curve with the Region III curve, as shown schematically in Figure 2. Because the Region III crack growth makes very little contribution to the time-dependent failure, it is treated as a vertical line in the development of the equations describing static, dynamic, and cyclic fatigue.[1]

The triangles in Figure 1 illustrate the results of the dynamic fatigue testing conducted at 850°C. For these data, the time was calculated by dividing the stress by the stressing rate. Note that the experimental dynamic fatigue data points are shifted to longer times because, in a dynamic fatigue test, the stress is not constant but increases linearly from zero to final fracture strength. The same relationship is reflected by the predicted static and dynamic fatigue curves. In the latter case, the curve was predicted from the static fatigue results by applying the slow-crack-growth parameters in Table 1 to the expression

$$[1-(S_f/\ S_i)^{N-2}] = [(N-2)/(2(N+1))]\ V(K_{IC})\,(Y\,S_i/K_{IC})^2\,(\tilde{\sigma}\,S_i)^N\ t \qquad (2)$$

where the stress, σ, now increases linearly with time. Again, at the point of failure, $S_f = \sigma$.

[1]The contribution of Region II slow crack growth to time-dependent failure is also ignored.

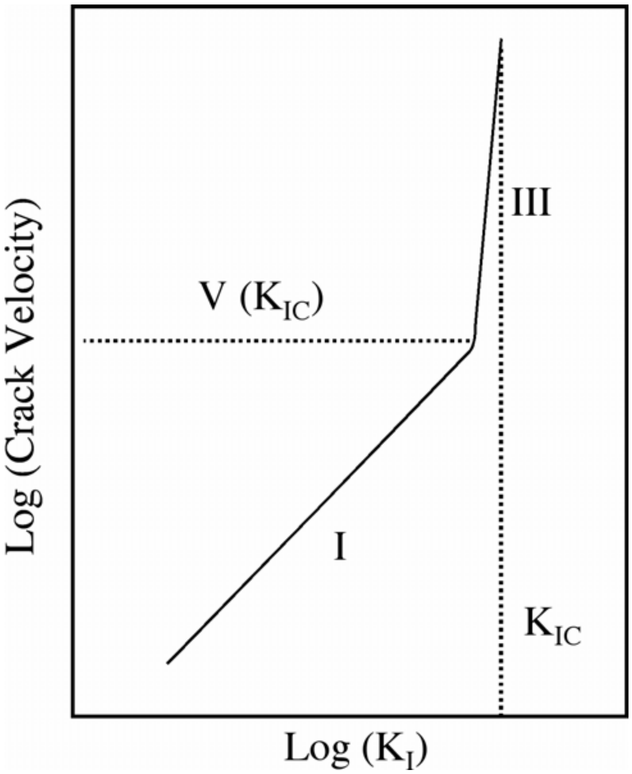

Figure 2. schematic representation of V-K_I behavior showing two regions of crack growth. Because the slope of Region III is very high, its contribution to time-dependent failure can be neglected. In this case, V (K_{IC}) represented the intersection of the Regions I and III curves.

Table 1. Crack growth parameters

Parameter	Value	Comments
Y	1.5	Based on surface cracks
K_{IC} (MPa m$^{1/2}$)	8	Based on vendors information
$V(K_{IC})$ (m/s)	5×10^{-6}	
S_i (MPa)	800	
N	25	

Conclusions

Subtask 11 (Annex II) activities have shown that existing slow-crack-growth models can be used to reconcile stress rupture, strength-stressing rate, and cyclic fatigue data.

References

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2. A. A. Wereszczak, H.-T. Lin, T. P. Kirkland, "Strength and Dynamic Fatigue of Silicon Nitride at Intermediate Temperatures," *J. Mater. Sci.* **37**, 2669–2684 (2002).

3. M. K. Ferber and M. G. Jenkins, "Evaluation of the Strength and Creep-Fatigue Behavior of a HIPed Silicon Nitride," *J. Amer. Ceram. Soc.* , **75**(9), 2453–62 1992.

FY 2003 Publications/Presentations

Mechanical and Thermal Fatigue of Silicon Nitride Within IEA Subtask 11, Japanese Final Report, January 2003.

International Energy Agency Implementing Agreement For A Programme Of Research And Development On Advanced Materials For Transportation Applications, Annual Report for Fiscal Year 2002, February 2003.

Implementing Agreement For A Programme Of Research And Development On Advanced Materials For Transportation Applications, End-Of-Term Report, February 2003.

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"United States Activities in Subtasks 11 and 13," presented at a working group meeting held in conjunction with the 27th annual Cocoa Beach Conference and Exposition on Advanced Ceramics and Composites, Cocoa Beach Florida, January 29, 2003.

C. Mechanical Property Test Development

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Contractor: Oak Ridge National Laboratory, Oak Ridge, Tennessee
Prime Contract No: DE-AC05-00OR22725

Subcontractor: National Institute for Standards and Technology, Gaithersburg, Maryland

Objectives

- Develop mechanical test method standards in support of the Propulsion Systems Materials Program. Ensure that test method development meets the needs of the DOE engine community and considers the general U.S. structural ceramics community, as well as foreign laboratories and companies.

Approach

- Investigate, refine, and standardize test methods relevant to ceramic materials and heavy vehicle propulsion applications.
- Conduct round-robins as necessary.
- Create standard reference materials (SRMs) to support the test methods and materials specifications.
- Have procedures standardized by the American Society for Testing and Materials (ASTM) and/or the International Organization for Standardization (ISO).

Accomplishments

- In FY 2003, finished SRM 2831 for Vickers Hardness of Ceramics and Hard Metals. This SRM complements ASTM and ISO test methods standards for Vickers hardness of hard materials and new material specifications standards, such as ASTM F 2094 for silicon nitride for ball bearings.
- Completed a major new study on the detection and characterization of grinding cracks in ceramics and published a comprehensive report. We now have a much better understanding of how grinding can affect the strength and reliability of ground ceramic test pieces and components. We also have a much better understanding of how to control the grinding damage.
- Updated and refined several ASTM standards, including ASTM C 1211 for high-temperature flexural strength and C 1322 for fractographic analysis.

Future Direction

- Evaluate and refine new test methods, including flexural strength testing of rod-shaped specimens and diametral compression strength of pill-shaped specimens. Both of these test configurations are highly relevant to many small engine parts, such as valve train or fuel injector components.
- Help to organize a new Applications Subcommittee for the ASTM Committee C-28, Advanced Ceramics. In the past, that committee focused on generic property standards, but the committee will now venture into more component- or application-specific standards.

Introduction

Sixteen formal standard test methods have been created as a result of work in this project. These include methods for ceramics such as flexural strength, elastic modulus, Weibull statistical analysis, fractographic analysis, and fracture toughness. As a direct result of this work, there has been a dramatic improvement in test data quality and reliability in the structural ceramics field. Databases are now more reliable and interchangeable. These developments have enhanced the credibility of the new materials and aided their commercial implementation in advanced engines. Engineers and designers are more apt to use the new materials if the materials are supported by good data generated by standard test methods. For example, formal standards have aided the recent commercial utilization of ceramic materials as ball bearings, cam rollers, and even human surgical implants.

Approach

New methods are devised, or more commonly, existent laboratory scale test methods are investigated, refined, and standardized. Often some preliminary work has shown that a particular test method has promise, but critical details need to be refined, or interlaboratory tests need to be conducted to verify within-laboratory repeatability or between-lab reproducibility. Comprehensive literature reviews and error analysis are key steps in the development of any formal standard.

SRMs have an important role in supporting overall standardization. An SRM is a well-characterized material produced in quantity to improve measurement science. It is certified for specific engineering, chemical, or physical properties and is issued by NIST with a certificate that reports the results of the characterization and indicates the intended use of the material.

Results

Hardness and SRM 2831 Finished

In FY 2003, we completed SRM 2831, Vickers Hardness of Ceramics and Hard Metals. This SRM is a tungsten carbide disk with certified hardness and with indentations made by NIST that may be measured by users. Users may also make their own new indentations to verify correct operation and make measurements with their own equipment and personnel. Hardness is an important attribute of ceramic material, and as Table 1 shows, there are two SRMs to support the two most common test methods to measure hardness.

Grinding Crack Evaluation Completed

Nearly all ceramic components, such as those shown in Figure 1, need expensive finish grinding to final dimensions. Remnant grinding cracks may affect the reliability of ceramic components in engines. Hence, it is important to characterize these potential strength-limiting flaws and manage their size and severity. Optimization of grinding procedures and reduction of machining costs

Table 1. Standard reference materials that are now available from NIST to support ASTM and International Organization of Standardization test method standards and materials specifications. All are available off the shelf.

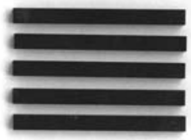
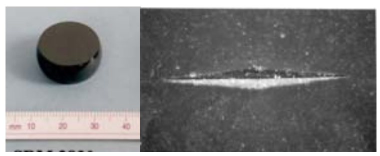
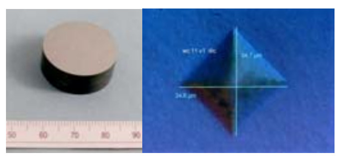
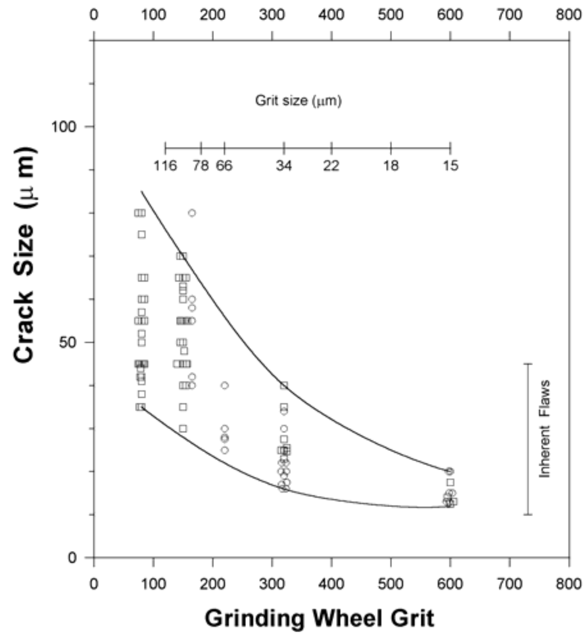
SRM	Images
# 2100 Fracture Toughness of Ceramics, K_{Ic}	
# 2830 Knoop Hardness of Ceramics	
# 2831 Vickers Hardness of Ceramics and Hardmetals	

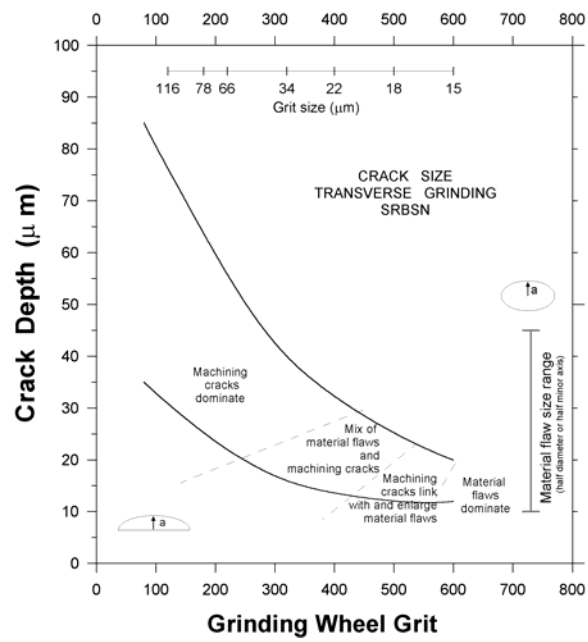
Figure 1. Ceramic engine components that require finish grinding.

are crucial in determining whether a ceramic component will be cost-effective.

A NIST Ceramic Machining Consortium program was completed in December 2001. Follow-on analysis and intensive fractographic examination of hundreds of Ceradyne sintered reaction-bonded silicon nitride (SRBSN) specimens continued until June 2003, when a major NIST special publication on the findings was published. A short condensed article was written for the *Ceramic Engineering and Science Proceedings* and will be presented at the Engineering Science Division meeting of the American Ceramic Society in Cocoa Beach, Florida, in January 2003. Two major journal articles



(a)



(b)

Figure 2. The depth of machining cracks created by surface grinding SRBSN silicon nitride primarily depends upon the wheel grit size of the grinding wheel. Figure (a) shows the data from individual crack size readings and (b) shows the overall trend and interactions with the material's inherent internal flaws.

were prepared and submitted to *Machining Science and Technology* in December and January 2003. Preprints have been sent to key parties in the United States.

We also learned that sometimes a single severe grit in a diamond wheel may control performance. A simple grinding crack damage map for SRBSN is shown in Figure 3. This drawing has been distributed widely to machinists and engineers to help them better appreciate the depth of grinding damage that is likely to be left in ground parts. Several different silicon nitrides were studied, and the results were compared with all known published data for silicon nitride. A stunning finding was that silicon nitrides with enhanced fracture toughness actually develop deeper machining cracks than do less tough nitrides. This seemingly paradoxical result is explained in the new manuscripts. We are also conducting detailed R-curve characterization of the SRBSN in collaboration with Prof. J. Rödel at the Technical University at Darmstadt, Germany.

Fractographic techniques to recognize machining crack damage on fracture surfaces have been made much easier. This new know-how has been transferred to the general community via a series of publications and revisions to the ASTM fractography standard C 1322. The nature of the machining damage is now much better understood. New damage maps for machining damage in silicon nitride have been constructed. Simplified versions will be sent to key machine shops to help them understand the nature of machining cracks from grinding.

New ASTM Applications Created

We helped ASTM Committee C 28, Advanced Ceramics, create a new subcommittee on applications. The committee had done a good job in preparing generic standard test methods since its inception in 1986, but interest in generic properties work was waning. Many of the key properties have already been standardized. Industry wants test methods and standards for specific components and applications. The new applications subcommittee will craft such standards and materials specification standards. The latter have already been prepared in ASTM by committees F-34 on Bearings and F-04 on Surgical and Medical Devices, based in part on Committee C-28's generic standards.

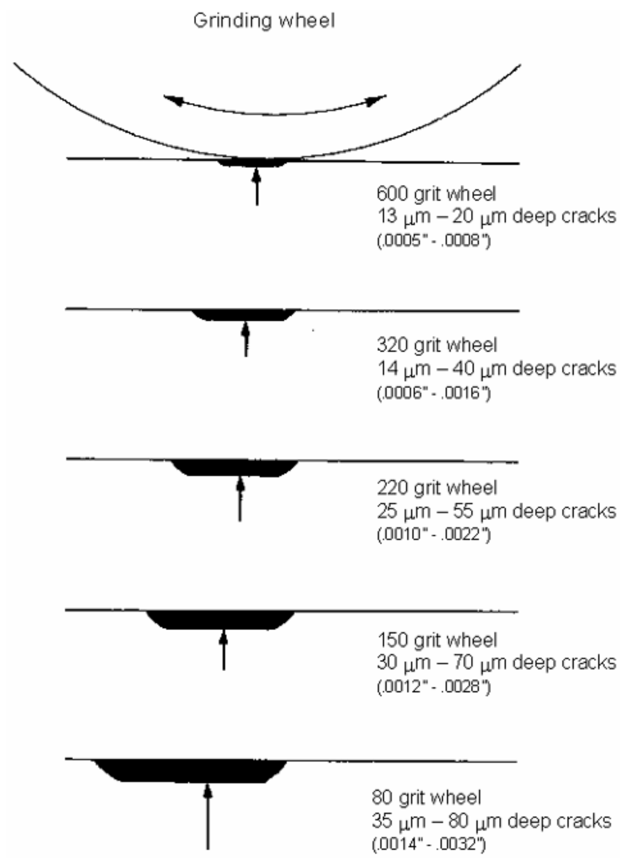


Figure 3. The depth of cracks (dark regions) created by surface grinding SRBSN silicon nitride primarily depends upon the wheel grit size of the grinding wheel.

ASTM Standards Revised and New ISO Standards Advanced

During FY 2003, we took many of the lessons learned from preparation of the SRMs, the ceramic grinding study, and other sources and made major revisions to the ASTM standards for fractographic analysis (C 1322), flexural strength at room temperature (C 1161), and flexural strength at elevated temperatures (C 1211). Revisions to the hardness standards are under way as well. Substantial progress was made on moving test methods for elevated temperature flexural strength (ISO 17565) and fracture toughness (ISO 18756) through the IOS system.

Conclusions

Step by step, we are building a national and international standards infrastructure to facilitate the commercial utilization of new advanced materials in engine applications. The generic test method standards developed to date have proved to be so practical, reliable, and versatile that they are now being used to support a wide range of applications, including surgical implants in humans and ceramic military body armor.

FY 2003 Publications/Presentations

1. G D. Quinn, K. Xu, J. A, Salem, and J. J. Swab, "SRM 2100: the World's First Fracture Toughness Reference Material," submitted to the Eighth International Conference on the Fracture Mechanics of Glasses and Ceramics, Houston, February 2003.

2. J. A. Salem, G. D. Quinn, and M. G. Jenkins, "Measuring the Real Fracture Toughness of Ceramics –ASTM C 1421," submitted to the Eighth International Conference on the Fracture Mechanics of Glasses and Ceramics, Houston, February 2003.

3. G. D. Quinn, "Weibull Strength Scaling for Standardized Rectangular Flexure Specimens," *J. Am. Ceram. Soc.*, **86(**3), 508–510 (2003).

4. G. D. Quinn, "Weibull Effective Volumes and Surfaces for Cylindrical Rods Loaded in Flexure," *J. Am. Ceram. Soc.*, **86**(3), pp. 475–478 (2003).

5. G. D. Quinn, L. K, Ives, and S. Jahanmir, *On the Fractographic Analysis of Machining Cracks in Ground Ceramics: A Case Study on Silicon Nitride,* NIST SP 966, National Institute of Science and Technology, May 2003.

6. G. D. Quinn, L. K, Ives, and S. Jahanmir, "Machining Damage Cracks: How to Find and Characterize Them by Fractography," to be published in *Ceram. Eng. Sci Proc.*, **24**(3) or (4), (2003).

7. G. D. Quinn, L. K, Ives, and S. Jahanmir, "On the Nature of Machining Cracks in Ground Ceramics: Part I: SRBSN Strengths and Fractographic Analysis," submitted to *Machining Science and Technology*.

8. G. D. Quinn, L. K, Ives, and S. Jahanmir, "On the Nature of Machining Cracks in Ground Ceramics: Part II: Comparison to Other Silicon Nitrides and Damage Maps," submitted to *Machining Science and Technology*.

9. G. D. Quinn, L. K. Ives, and S. Jahanmir, "Fractography Reveals Machining Cracks," *Bul. Amer. Ceram. Soc.*, **82**(7), 11 (2003).

10. G. D. Quinn, J. J. Swab, and M. J. Motyka, "Fracture Toughness of a Toughened Silicon Nitride by ASTM C 1421," *J. Am. Ceram. Soc.*, **86(**6), 1043–1045 (2003).